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USA
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To:
Ms. Sue Leeming & Ms. Abigail Hendershott
Michigan Department of Environmental Quality
Remediation & Redevelopment Division
Grand Rapids District Office
350 Ottawa Ave NW, Unit 10
Grand Rapids, MI 49503-2341

CC:
John Cuthbertson, AECOM

From:
Robert Kennedy, AECOM

Date:
March 16, 2018

Memo

Subject: House Street - North Kent Disposal Investigation Site PFAS Three Laboratory Split Sample Well Water Data Analysis, Kent County

Split samples were collected from untreated well water at three addresses within the House Street - North Kent Disposal Investigation Site as part of an effort by MDEQ to evaluate the comparability of per- and polyfluoroalkyl substances (PFAS) analytical data between three laboratories being used in the project. Two of the laboratories (ALS-Kelso and Eurofins-Lancaster) are being used by GZA for Wolverine, and the third laboratory (Vista) is being used by AECOM for MDEQ. All of these laboratories are using LC/MS/MS isotope dilution technique modified methods to quantify PFAS target analytes, and incorporate similar basic features such as using weak anion exchange solid phase extraction media (WAX SPE) for extraction, electrospray ionization tandem mass spectrometry conditions and transition ion choice, association of native and labeled isotopes for isotope dilution analysis (IDA), calibration standards source (Wellington), and quality control criteria derived from either EPA Method 537, the DoD Quality Systems Manual Revision 5.1 (QSM 5.1) guidance on PFAS analysis by isotope dilution, or in-house statistical performance criteria.

Locations were selected that provide a range of concentrations typical of the site, from low to middle and high, with reportable detections of PFOS, PFOA, and associated PFAS target analytes. Reports from the three laboratories were subjected to full data validation (Level 4 or Stage 4B) including the review of raw data with recalculations of select laboratory results. In the absence of published method specific data validation guidance from EPA for PFAS analysis, the *National Functional Guidelines for Organic Superfund Methods Data Review* (January 2017), the *National Functional Guidelines for High Resolution Superfund Methods Data* (April 2016), and the laboratory Standard Operating Procedures for PFAS analysis in water by isotope dilution technique were used to provide rules and criteria for data validation.

Data validation memoranda are attached for each lab report reviewed. Qualifiers were added to results where necessary based on the data validation. No data were rejected and all results are considered usable for decision making purposes.

In order to understand the magnitude and significance of differences between datasets, results were compiled into **Table 1**, attached. Results for analytes not reported by all three labs (N-EtFOSE, N-MeFOSE, PFHxDA, and PFODA) were excluded, but these compounds were also nondetect at all locations and therefore not relevant to the inter-laboratory comparison. Relative standard deviations (RSD) for each analyte were calculated to assess inter-laboratory precision (light blue highlighted columns). Colors were added to the detected values

text to indicate in each location set the highest (red), middle (blue), and lowest (green) result for each analyte. Cases where target analytes were detected in two or more labs at each location are highlighted yellow.

In general, RSD values for most analytes were less than 30%, which is within the range of expected analytical variation based on uncertainty due to method performance criteria such as calibration verification. The mean RSD for all analytes at the Ex. 6 Personal Privacy (PP) location was 29%, the mean RSD at the Ex. 6 Personal Privacy (PP) location was 19%, and the mean RSD for the Ex. 6 Personal Privacy (PP) location was 14%.

A few specific analytes had significantly higher RSDs, and closer inspection of the raw data confirms there are methodological reasons that account for some of the inter-laboratory bias observed in these split results. As part of the data validation and review of the laboratory SOPs, followed up by resubmittal questions to the three laboratories for confirmation, a description of some laboratory procedural differences was compiled in **Table 2**, attached. The most potentially significant differences fall into four groups: (1) PFAS target peak integration rules in samples; (2) standard peak integration and calibration procedures; (3) dilution practices with regard to the use of labeled standards and principles of isotope dilution; and (4) reporting conventions. These differences will be discussed below, with a bit of background information to explain them in context, and examples from the reviewed lab reports.

The laboratories have adopted different procedures with respect to branched and linear isomer peak integration. When PFAS compounds are produced by the electrochemical manufacturing process, such as the one used by 3M in their production of Aqueous Film Forming Foam (AFFF) and Scotchgard™ products, both linear and branched isomers are created for each PFAS compound produced. These isomers may (or may not) be separated by the laboratory LC/MS/MS conditions, depending on the columns used, eluant mixture, flowrates, and gradient conditions during the liquid chromatographic separation. Isomer separation is not a requirement of EPA Method 537 rev. 1.1 or the additional method performance criteria added by DoD in the Quality Systems Manual Revision 5.1 in Table B-15 for PFAS analysis. The rules governing linear and branched peak integration presented in these documents are subject to multiple interpretations. EPA became aware of this when reviewing UCMR 3 PFAS data and issued a guidance document about PFOA in particular (EPA 815-B-16-021). This guidance instructs laboratories to integrate both the branched and linear isomers together in the PFOA chromatogram, but quantify them using a linear isomer only standard calibration for response factors, and qualitatively identify peaks as branched or linear using "qualitative/semi-quantitative" PFOA mixed isomer standards. The EPA Method 537 Rev.1.1 recommends using quantitative mixed isomer standards for PFOS, PFHxS, NetFOSAA and NMeFOSAA calibration only, which were the only quantitative mixed isomer PFAS target analytes commercially available at the time the method was written. Only the linear isomer standards are available for most of the other PFAS target compounds. In the absence of explicit instructions about what to do for the other PFAS compounds laboratories have adopted sometimes different practices in their isotope dilution based SOPs. None of these practices are "wrong" or "right" because they are not specifically required or prohibited by guidance documents, but the differences do have observable consequences.

The ALS-Kelso laboratory quantified all the branched and linear peaks for all the PFAS targets when detected. The Vista and Eurofins-Lancaster labs only integrated the combined branched and linear isomer peaks for PFOS, PFHxS, and PFOA because these have commercial mixed isomer standards. This may explain part of the reported result differences and >25% RPDs for compounds such as PFHpA, PFHpS, and PFNA; however it should be noted that ALS-Kelso did not always report the highest result for compounds, and that for other targets such as PFBA, PFBS, and PFPeA where the integration practices were also different, the RSDs were generally only in the 11 to 21% range. The ratio of branched to linear peak area varies between different PFAS compounds, and can be altered by fate and transport processes, which greatly complicates the potential effect of the integration practice difference, therefore a simple uniform effect is not to be expected.

An additional complication in lab practice is the use of secondary or confirmatory ion transitions. These confirmatory ion sets are intended to be used, per the reference methods and DoD guidance, for qualitative target identification. The ALS and Eurofins labs do not provide extracted ion current profiles (EICPs) for these confirmatory ions, and have stated in responses to questions that specific criteria are not applied based on the confirmatory ion response to determine which peaks are integrated. Vista on the other hand has adopted specific criteria for peak signal to noise ratios and the comparison of standard and sample peak retention time

matches for PFOS, PFOA, and PFHXS. These criteria can lead them to exclude specific peaks from integration if not confirmed in the reference standard patterns, as described in **Table 2**, and presented in **Figure 1**, which could bias the Vista results low relative to other labs; however, the outcome in reported result differences is not uniform for all samples and all analytes in this group, as can be seen in the **Table 1** result colors.

Regarding the differences in laboratory calibration practices, ALS-Kelso is using response factors from the linear isomer only for all compounds except PFOS and PFHxS where they calibrate branched and linear isomers combined using the Wellington mixed isomer standard. Both Eurofins-Lancaster and Vista are calibrating with response factors based on the linear isomer only for all PFAS targets and use the same mixed isomer standards of PFOS, PFOA, and PFHxS only for qualitative peak pattern recognition (but not in the same way, as described above). The effect of this calibration difference is unknown because the labs do not provide enough information in the full data packages or SOPs to evaluate the difference. Anecdotal information from the laboratories, as well as the peer reviewed literature, indicates there are differences in the response factors for branched and linear isomers, but the magnitude is unknown. It is also worth pointing out that the ALS-Kelso lab has a more limited calibration standard range (0.01 to 20 ng/mL) than either Vista (0.25 to 100 ng/mL) or Eurofins-Lancaster (0.2-100) ng/mL, and that the extracted internal standard concentration (ES) is 5 ng/mL for both Kelso and Eurofins- Lancaster, but 12.5 ng/mL for Vista. These differences can affect the need for dilutions, which can in turn affect combined run result datasets, however the direction of this bias (if any) is unknown and the ES sample spiking and dilution practices described below probably have a greater impact.

Regarding the difference in laboratory dilution practices and use of ES, both ALS-Kelso and Vista use true isotope dilution technique consistent with the HRMS IDA methods such as EPA 1613B and EPA 1668C. The ES is added only before extraction and final quantitation is based on the ratio of labeled and target ion responses in standards and samples, per a formula similar to that provided in EPA Method 8290A. This means that the maximum dilution is limited by the spiked ES concentration and the instrumental ability to provide adequate S/N for the ES in the diluted extract. It should be noted that ALS-Kelso uses a nominal ~200 ng/L ES spike for 50mL samples, where Vista uses a nominal ~50 ng/L ES spike for 250 mL samples, and that both labs used the same ES lot for calibration and sample spiking. When the initial extract ES would be over diluted to quantify the highest concentration target analytes, then a smaller aliquot of another sample containers must be re-spiked and re-extracted for the highest reported dilutions. The Eurofins-Lancaster lab, on the other hand, adds additional ES to all initial extract dilutions so as to maintain a constant ES concentration in the analyzed extract. This compensates for the ES dilution and obviates the need to re-extract a smaller aliquot; however it violates the principle of isotope dilution in the EPA HRMS methods because the final reported result is not recovery corrected for all the effects of extraction, cleanup, and analysis. This may have a very significant effect on reported results if matrix effects from preparation or analysis are substantial. Any volumetric or concentration errors in re-spiking could also affect the result because results are adjusted by the ES, however the net effect is that the ES becomes more like an internal standard (IS) added post extraction. The net bias on the split sample results of these combined effects is difficult to estimate, but could be significant, especially for the high concentration 1850 House Street sample which required dilution. Another difference between the labs with regard to ES-target associations which affects only target PFHpS, is that for ES the ALS-Kelso laboratory uses 18O-PFHxS, but Eurofins-Lancaster uses 13C3-PFHxS, and Vista uses 13C2-PFOA. True isotope dilution, where the ES is an isotopically labeled version of the associated target compound is not currently possible for PFHpS because labeled PFHpS is not commercially available. This forces the labs to find an alternative related labeled standard compound to quantify PFHpS. Any chemical differences in the behavior of the native target and ES could affect the quantitation.

Regarding the laboratory reporting conventions, ALS-Kelso is using the method reporting limit (MRL) of ~4 ng/L and extracted a nominal volume of 60mL per sample. Estimated results below the MRL and above the MDL are not reported by ALS-Kelso for this project. Eurofins extracted a nominal 250mL per sample and reported all detections down to the nominal MDL and J flagged the estimated values between the LOQ and MDL. Vista also extracted 250mL per sample, but used the DoD reporting convention of LOQ/LOD/DL, where results below the LOQ and above the DL are J flagged, and the LOD value is used as the reporting detection limit (RDL = value behind the < for nondetects in **Table 1**). This mixture of conventions, combined with different nominal sample sizes, can easily create misunderstanding of the relative sensitivity of the various lab methods, which are all very similar at the instrument level, however in undiluted samples for nondetect results the RDLs appear to be

significantly different (ALS>Vista>>Eurofins). This analysis has focused on comparison of detected values because nondetects are not easily amenable to precision analysis, however it should be noted that detect/nondetect bias could be perceived simply due to the reporting conventions used by these three laboratories, regardless of actual method sensitivity.

In conclusion, we have evaluated the PFAS split sample results from all three laboratories and found the results all useable, based on data validation guidance and professional judgment, and comparable within the limits of analytical uncertainty typical of environmental methods. We also found evidence of potential systematic bias in the different procedures used by the three laboratories with regard to target PFAS peak integration rules, calibration procedures, and reporting conventions. Some of these procedural differences could be ameliorated by establishing project specific conventions for all participating laboratories to follow.

Figure 1 - Laboratory Chromatogram Examples

eurofins | Lancaster Laboratories

LC-MS/MS Analysis Report

Printed: 12/13/2017 4:02:00 PM
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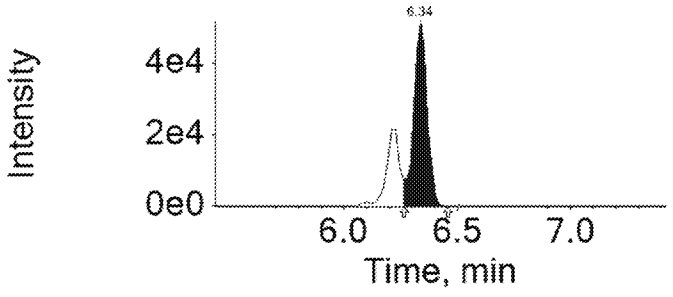

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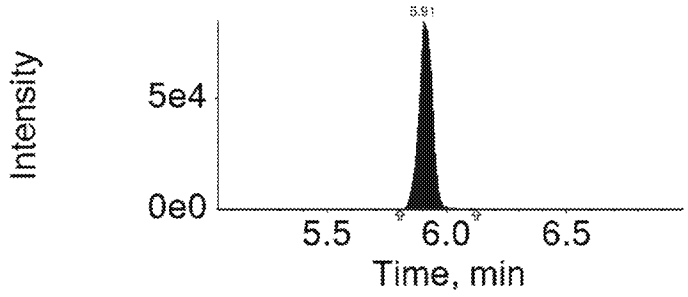
402.0 / 80.0



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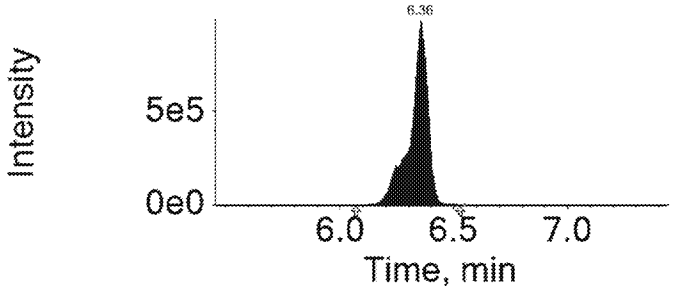
413.0 / 369.0



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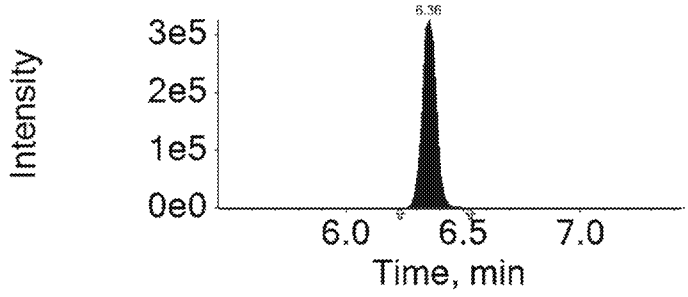

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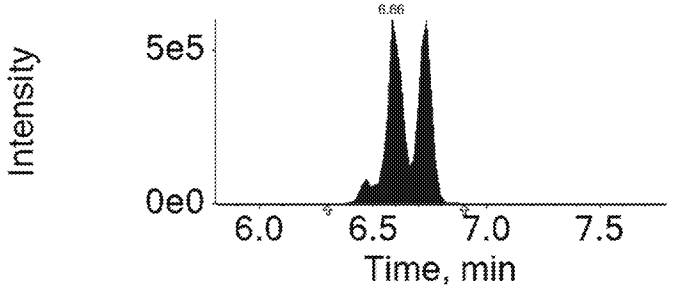

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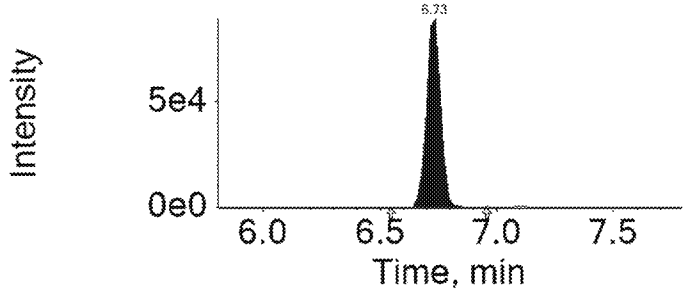

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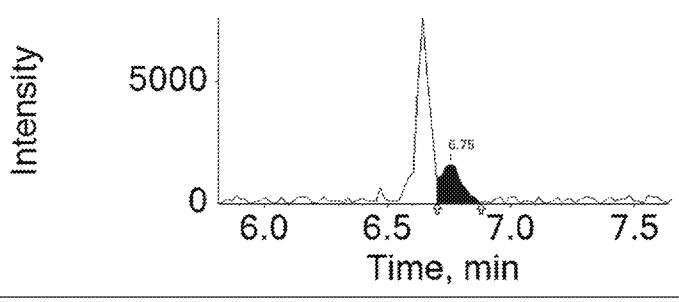

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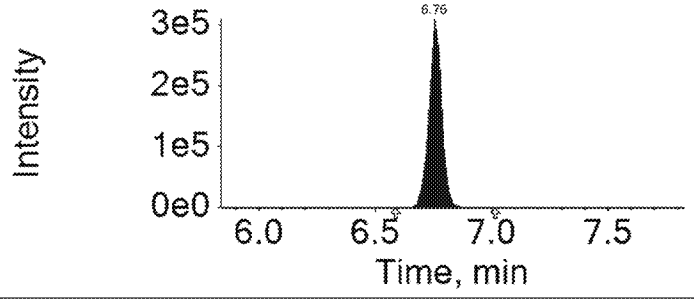

APPROVED
By MCD at 4:08 pm, 12/13/17

REVIEWED
By ucmr at 4:51 pm, 12/13/17

Insight Report

Printed at 12/22/2017 4:59:32 PM

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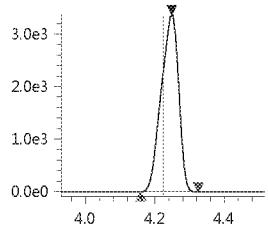

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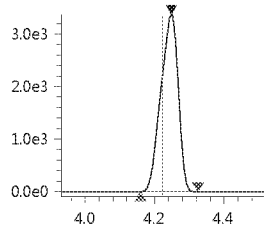

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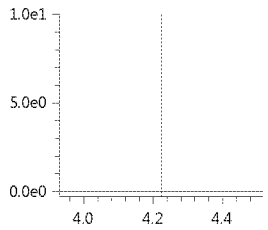

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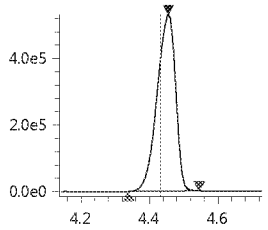

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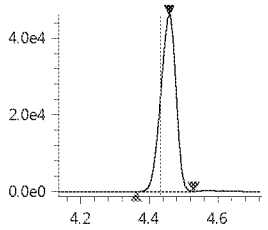

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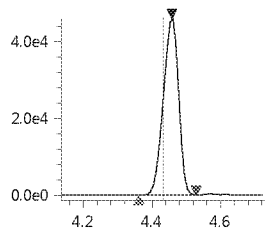

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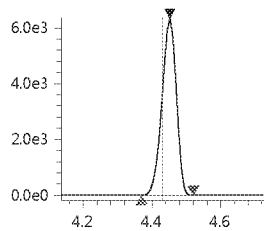

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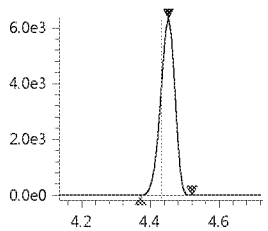

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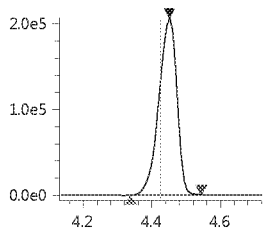

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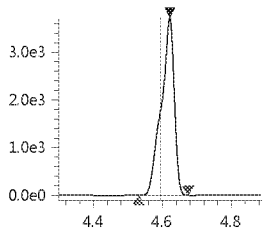

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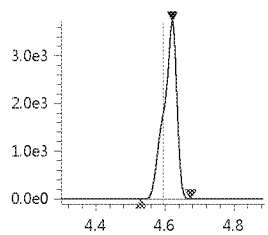

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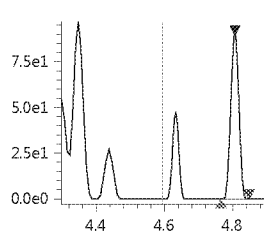

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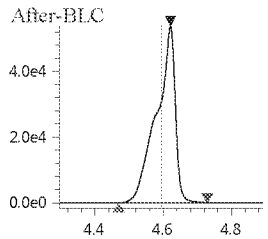

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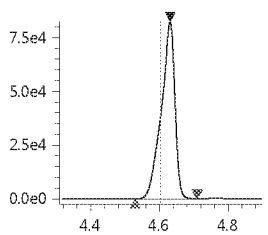

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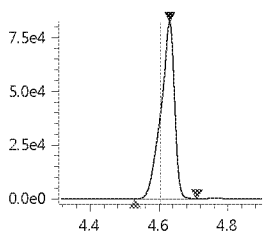

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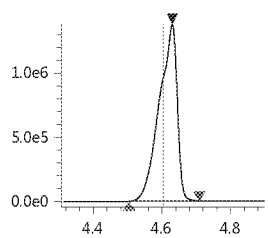

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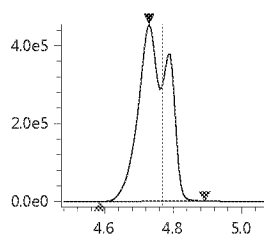

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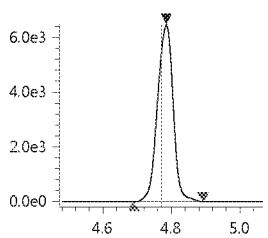

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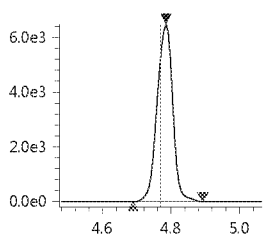

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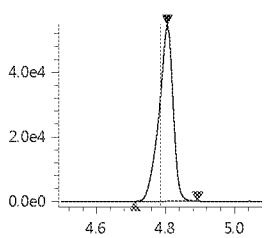

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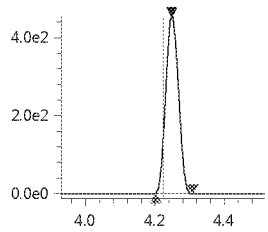
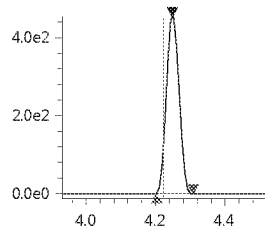
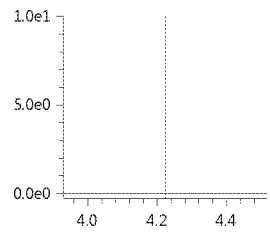


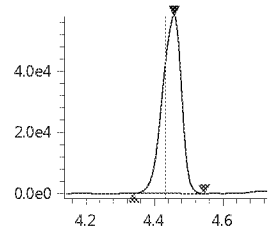
4_2-FTS-13C
Conc 0.0446
RT 4.249 Area 1165
Q 328.80>309.05 4.55e2



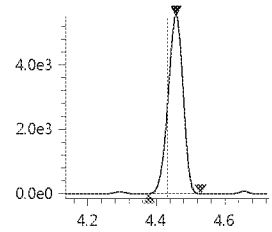
4_2-FTS_1
Conc ----
RT ---- Area ----
Q 327.00>307.05 0.00e0



PFHpA
Conc 49.9279
RT 4.457 Area 224947
Q 362.90>319.00 5.80e4



PFHpA-13C_IS
Conc 5.0000
RT 4.457 Area 16870
ISTD 366.90>322.10 5.51e3



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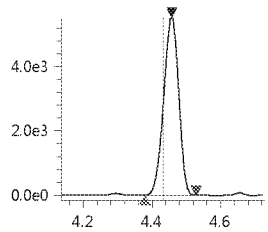
Conc 0.0446
RT 4.457 Area 16870
Q 366.90>322.10 5.51e3



PFHxS-18O_IS
Conc 5.0000
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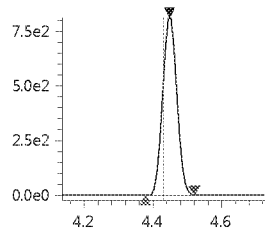
ISTD 402.70>84.05 8.12e2



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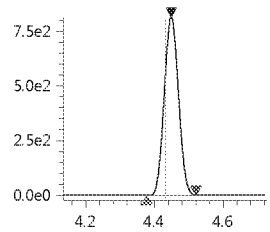
Q 402.70>84.05 8.12e2



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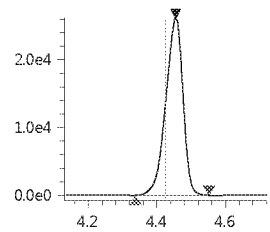
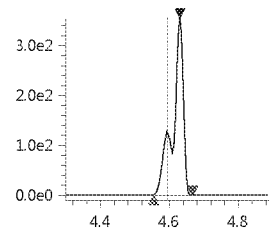
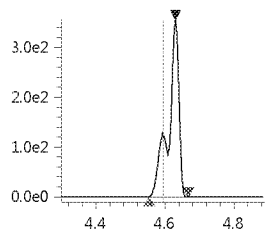
RT 4.455 Area 91569
Q 399.00>80.00 2.62e4



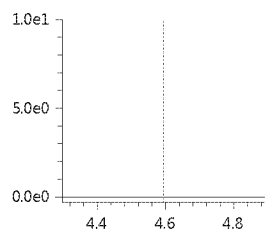
6_2-FTS-13C
Conc 0.0325
RT 4.630 Area 743
Q 428.90>409.00 3.56e2



6_2-FTS-13C_IS
Conc 5.0000
RT 4.630 Area 743
ISTD 428.90>409.00 3.56e2



6_2-FTS_1
Conc ----
RT ---- Area ----
Q 427.00>407.00 0.00e0



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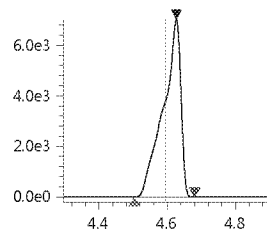
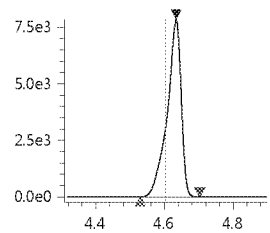
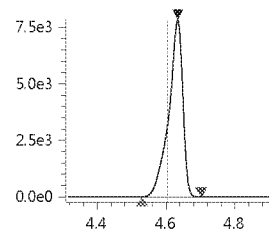
Conc 42.1424
RT 4.627 Area 25000
Q 449.00>80.15 7.02e3



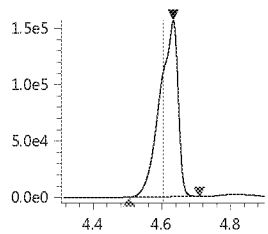
PFOA-13C_IS
Conc 5.0000
RT 4.634 Area 22099
ISTD 416.80>372.05 7.84e3



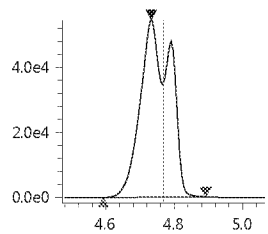
PFOA-13C
Conc 0.0468
RT 4.634 Area 22099
Q 416.80>372.05 7.84e3



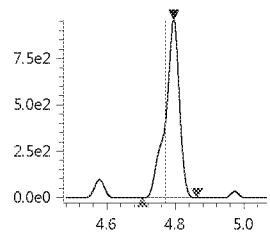
PFOA
Conc 192.7651
RT 4.633 Area 572652
Q 413.00>369.00 1.57e5



PFOS_1
Conc 583.9237
RT 4.733 Area 334313
Q 499.00>80.00 5.45e4



PFOS-13C_IS
Conc 5.0000
RT 4.795 Area 2724
ISTD 502.80>80.05 9.57e2



PFOS-13C
Conc 0.0546
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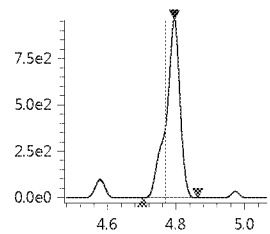
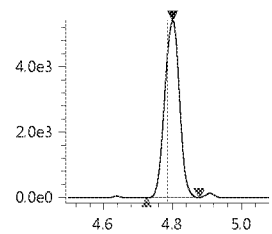
Q 502.80>80.05 9.57e2



PFNA-13C_IS
Conc 5.0000
RT 4.801 Area 16176
ISTD 467.80>423.00 5.43e3



Quantify Sample Report **MassLynx MassLynx V4.1 SCN945 SCN960**

Vista Analytical Labs

Dataset: U:\Q4.PRO\results\180110M3\180110M3_68.qld

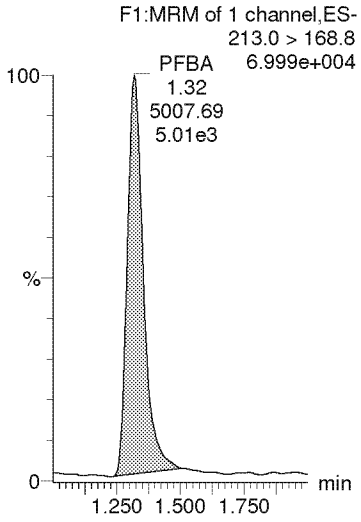
Rev'd: MM 1/12/18

Last Altered: Friday, January 12, 2018 15:44:03 Pacific Standard Time
Printed: Friday, January 12, 2018 15:47:20 Pacific Standard Time

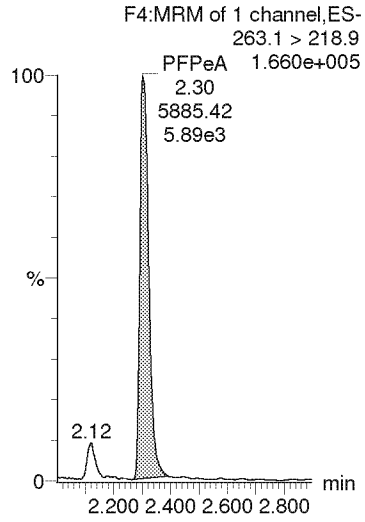
Method: U:\Q4.PRO\MethDB\PFAS_FULL_80C_010818C.mdb 11 Jan 2018 15:33:36
Calibration: U:\Q4.PRO\CurveDB\C18_VAL-PFAS_Q4_01-10-18-FULL-M3.cdb 11 Jan 2018 14:26:30

Name: 180110M3_68, Date: 11-Jan-2018, Time: 21:51:57, ID: 1701905-03RE1@10X WINF1712061655JLB 0.25, Description: WINF1712061655JLB

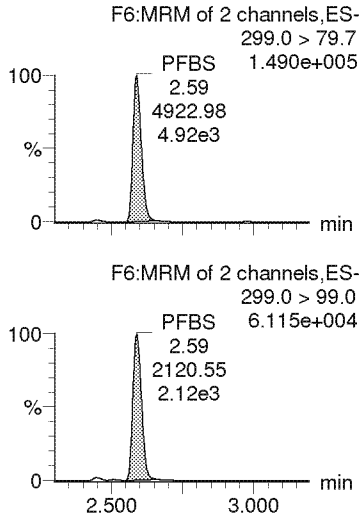
PFBA



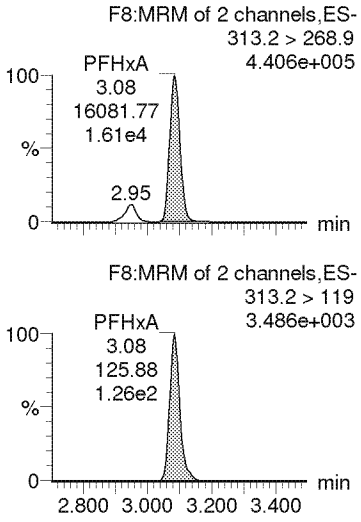
PFPeA



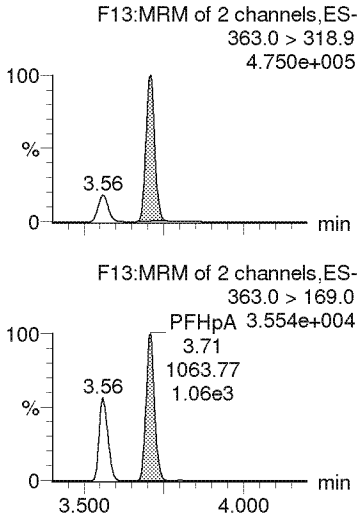
PFBS



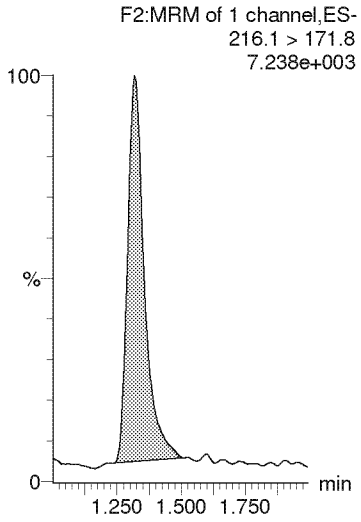
PFHxA



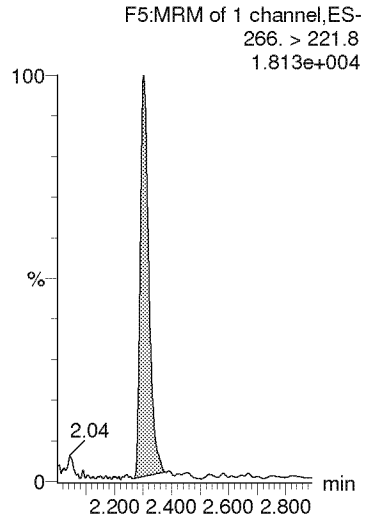
PFHpA



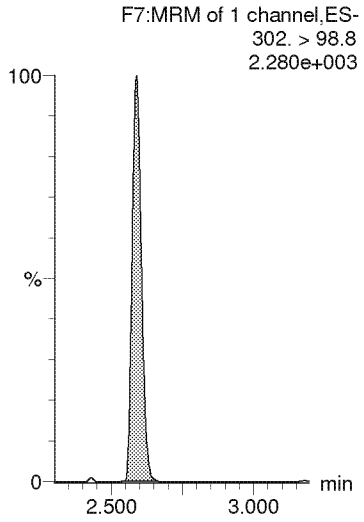
13C3-PFBA



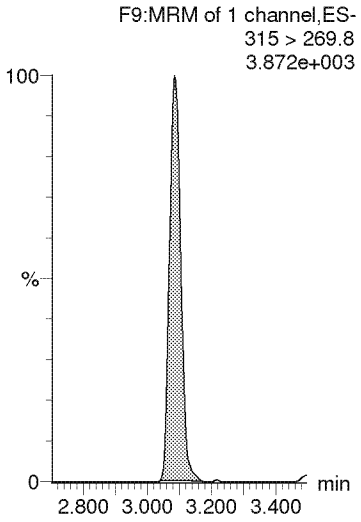
13C3-PFPeA



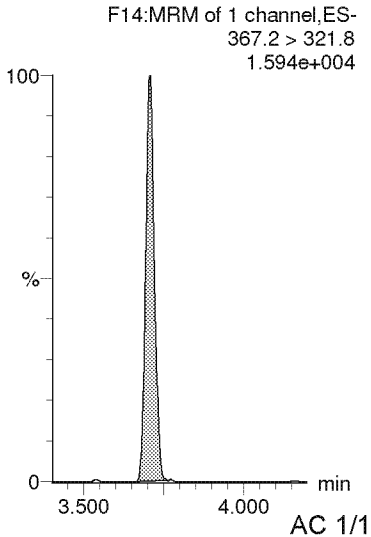
13C3-PFBS



13C2-PFHxA



13C4-PFHpA



AC 1/12/2018

Quantify Sample Report **MassLynx MassLynx V4.1 SCN945 SCN960**

Vista Analytical Laboratory

Rev'd: MM 1/12/18

Dataset: U:\Q4.PRO\results\180110M3\180110M3_67.qld

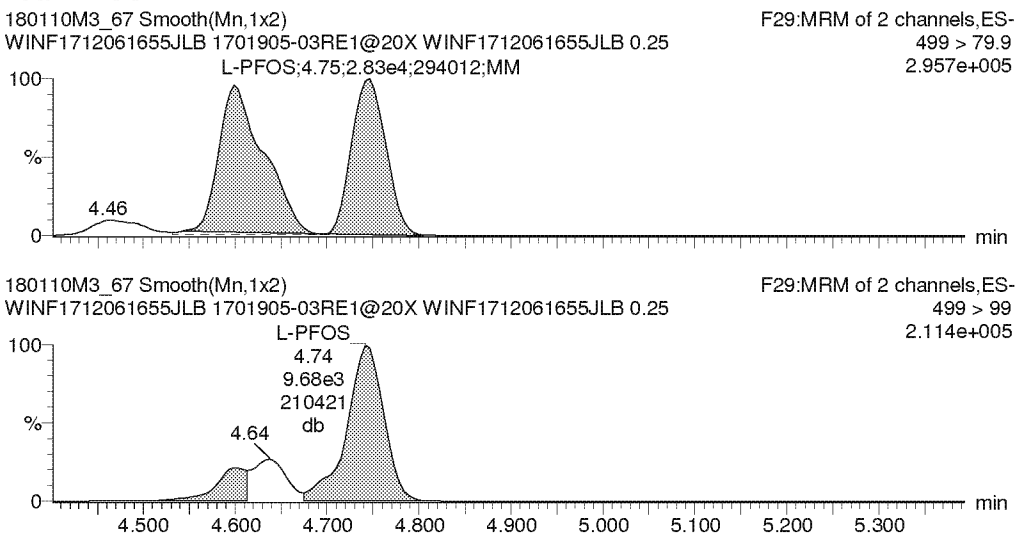
Vista Analytical Labs

Last Altered: Friday, January 12, 2018 16:06:46 Pacific Standard Time
Printed: Friday, January 12, 2018 16:16:27 Pacific Standard Time

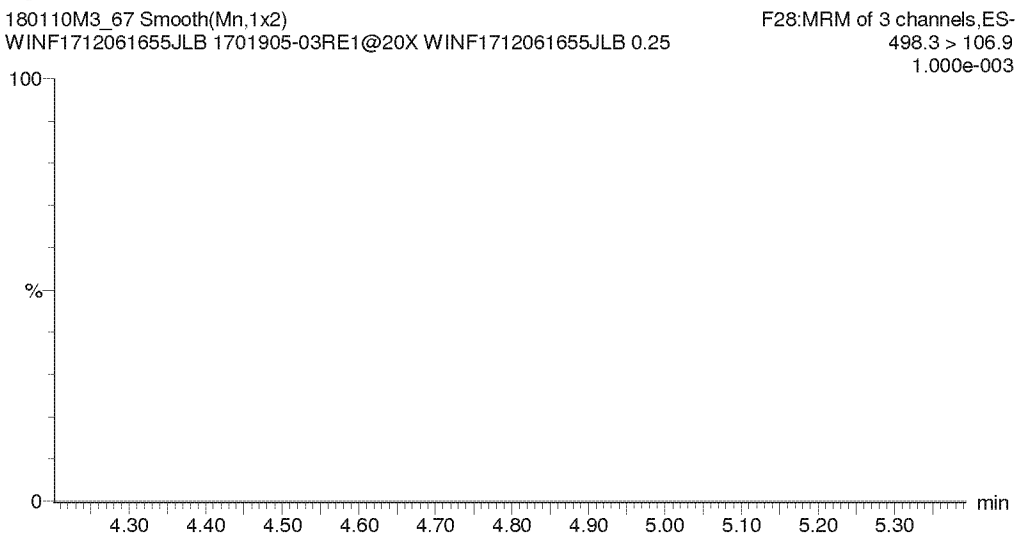
Method: U:\Q4.PRO\MethDB\PFAS_FULL_80C_010818C.mdb 11 Jan 2018 15:33:36
Calibration: U:\Q4.PRO\CurveDB\C18_VAL-PFAS_Q4_01-10-18-FULL-M3.cdb 11 Jan 2018 14:26:30

Name: 180110M3_67, Date: 11-Jan-2018, Time: 21:40:47, ID: 1701905-03RE1@20X WINF1712061655JLB 0.25, Description: WINF1712061655JLB

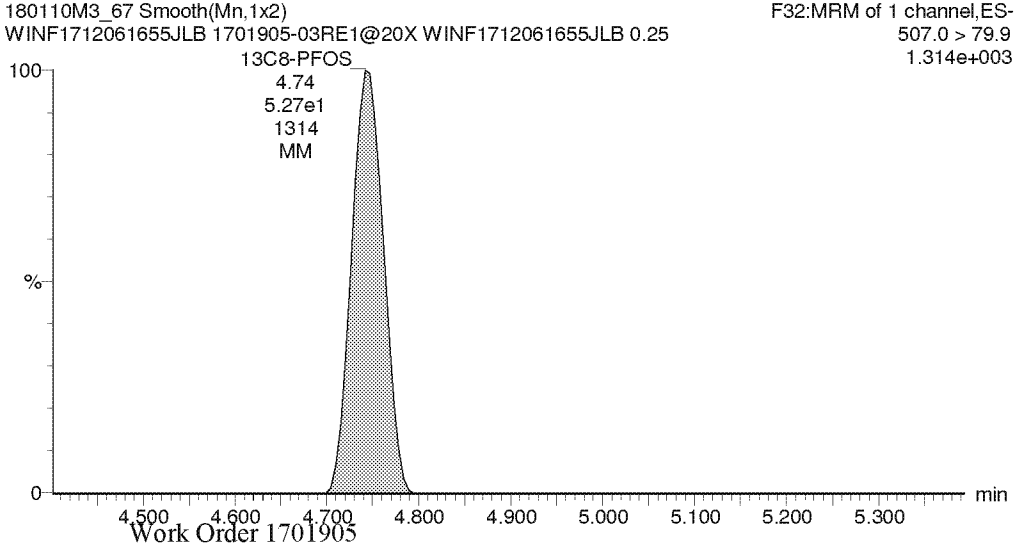
Total PFOS



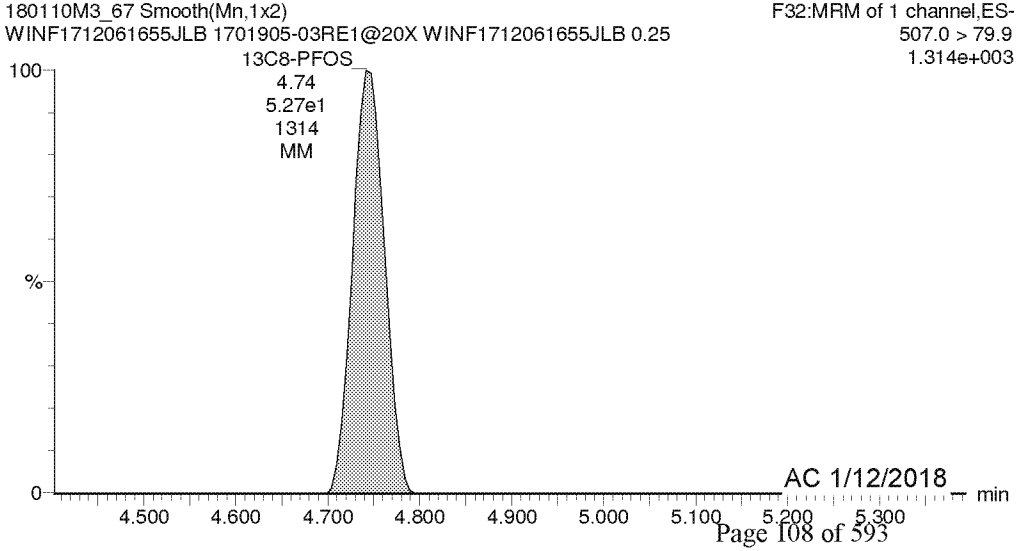
TCDA



13C8-PFOS



13C8-PFOS



AC 1/12/2018

Table 1. [redacted] Ex. 6 Personal Privacy (PP)

Ex. 6 Personal Privacy (PP)

	sys_loc_code													
	sys_sample_code													
	sample_date													
	lab_name_code													
	lab_sdg													
	lab_sample_id													
	sample_type_code		N	N	N	28.75%	N	N	N	19%	N	N	N	13.56%
Chemical name	PFAS abbv.	Units				RSD				RSD				RSD
6:2 Fluorotelomer sulfonic acid	6:2 FTS	ng/l	< 4.5	< 2.52	< 300	na	< 4.3	1.14	< 3	NA	< 4.3	< 2.45	< 3	NA
8:2 Fluorotelomer sulfonic acid	8:2 FTS	ng/l	< 4.5	< 2.52	< 200	na	< 4.3	< 2.45	< 2	NA	< 4.3	< 2.45	< 2	NA
Perfluorobutanoic acid	PFBA	ng/l	350	401	460	14%	< 0.6	8.1	11	21%	21	22.8	28	15%
Perfluorobutanesulfonic acid	PFBS	ng/l	1400	1240	1700	16%	38	33.8	46	16%	130	120	150	11%
Perfluorodecanoic acid	PFDA	ng/l	< 4.5	< 2.52	< 50	na	< 4.3	< 2.45	< 0.4	NA	< 4.3	< 2.45	< 0.4	NA
Perfluorododecanoic acid	PFDoDA	ng/l	< 4.5	< 2.52	< 30	na	< 4.3	< 2.45	< 0.3	NA	< 4.3	< 2.45	< 0.3	NA
Perfluorodecanesulfonic acid	PFDS	ng/l	< 4.5	< 2.52	< 50	na	< 4.3	< 2.45	< 2	NA	< 4.3	< 2.45	< 2	NA
Perfluoroheptanoic acid	PFHpA	ng/l	1600	914	1500	28%	22	16.7	23	16%	94	74.2	96	14%
Perfluoroheptanesulfonic acid	PFHpS	ng/l	1300	593	1200	37%	7	2.14	3	64%	120	99.9	90	15%
Perfluorohexanoic acid	PFHxA	ng/l	900	977	1400	25%	33	25.3	34	15%	82	64	78	13%
Perfluorohexanesulfonic acid	PFHxS	ng/l	5400	4260	6000	17%	110	93.8	160	28%	340	290	440	21%
Perfluorononanoic acid	PFNA	ng/l	29	8.43	< 40	78%	< 4.3	< 2.45	2	NA	< 4.3	0.492	0.9	41%
Perfluorooctanoic acid	PFOA	ng/l	5800	4360	6800	22%	62	51.5	89	29%	460	653	570	17%
Perfluorooctanesulfonic acid	PFOS	ng/l	18000	29800	28000	25%	4.9	1.26	4	56%	1200	1130	1500	15%
Perfluorooctane sulfonamide	PFOSA	ng/l	< 4.5	< 2.52	< 90	na	< 4.3	< 2.45	< 0.3	NA	< 4.3	< 2.45	< 0.3	NA
Perfluoropentanoic acid	PFPeA	ng/l	450	454	550	12%	8.1	8.12	10	12%	27	26.8	35	16%
Perfluorotetradecanoic acid	PFTeDA	ng/l	< 4.5	< 2.52	< 30	na	< 4.3	< 2.45	< 0.3	NA	< 4.3	< 2.45	< 0.3	NA
Perfluorotridecanoic acid	PFTrDA	ng/l	< 4.5	< 2.52	< 30	na	< 4.3	< 2.45	< 0.3	NA	< 4.3	< 2.45	< 0.3	NA
Perfluoroundecanoic acid	PFUnDA	ng/l	< 4.5	< 2.52	< 40	na	< 4.3	< 2.45	< 0.4	NA	< 4.3	< 2.45	< 0.4	NA

Table 2. Comparison by Lab for Issues that May Impact Quant for Splits

Laboratory	ICAL Range	SPE cartridge	Quant and confirmation ions?	Branched and Linear in Calibration stds	Sample by vol or mass	Typical sample volume for analysis	Spike conc. of ES in samples	Spike conc. of Injection IS or Recovery Std. (RS) . in samples	Sample DF	Additional ES added for dilutions?	IS association for target compounds not quanted by IDA with labeled analog	Extract storage	Reporting convention
Lancaster	**All target compounds except as noted below (ng/L):** 0.200, 0.600, 2.00,8.00, 20.0, 50.0, 100 **PFBS**: 0.170, 0.530, 1.77, 7.08, 17.69, 44.22, 88.45 **4:2 FTS**: 0.580, 1.980, 4.900, 10.04, 12.49 **PFPeS:** 0.190, 0.560, 1.88, 7.500, 18.76, 46.9, 93.8 **PFHxS:** 0.190, 0.570, 1.89, 7.56, 18.910, 47.28, 94.55 **6:2 FTS:** 1.300, 2.090, 5.210, 10.43, 13.04 **PFHpS:** 0.190, 0.570, 1.900, 7.610, 19.03, 47.58, 95.15 **PFOS:** 0.190, 0.570, 1.910, 7.65, 19.12, 47.8, 95.6 **8:2 FTS:**1.320, 2.110, 5.270, 10.54, 13.170 **PFDS:** 0.190, 0.580, 1.93, 7.700, 19.260, 48.150, 96.30 ES: ES are 5.0 except 13C8-PFOS, 13C9-PFNA (4.78) in CAL1-CAL7	From lab SOP: Solid phase extraction (SPE) cartridge – Waters Sep-Pak C18 6 cc Vac Cartridge, 500 mg Sorbent per Cartridge, 55-105 µm Particle Size Cat. No. [WAT043395] Per email response 3/1/2018: the lab uses a WAX cartridge.	Per email response (2/26/2018): confirmation ions are not monitored for all compounds need clarification for what this means; response pending.	L +B standard analyzed and used only for identification. Quantitation based on linear response only for all targets. Ex. 6 Personal Privacy (PP) **[SDG 1883885]:** Branched isomers also present for PFHpA, PFHpS, PFNA but only included in integration for PFHxS, PFOS and PFOA. **[SDG 1876323]:** Ex. 6 Personal Privacy (PP) **11/14:** Branched isomers also present for PFPeA, PFHxA, PFHpA, PFHpS, and PFNA but only included in integration for PFHxS, PFOS and PFOA . Ex. 6 Personal Privacy (PP) **1/14:** Branched isomers also present for PFPeA, PFHxA, PFHpA, and PFHpS but only included in integration for PFHxS, PFOS and PFOA.	mass	250 ml [per email response 3/1/2018: The lab uses the entire volume and rinses the container which is then extracted]	(The lab responded that the ES used to spike the samples is not necessarily the same lot that was used to spike the calibration stds. The ES is checked against a current calibration prior to use) **For undiluted samples: all ES except as noted below:** 18.761 ng/ml **13C3-PFHxS:** 17.748 ng/ml **13C2-6:2-FTS:** 17.823 ng/ml **13C8-PFOS**: 17.936 ng/ml **13C2-8:2FTS:** 17.973 ng/ml	13C3-PFBA: 5.00 ng/ml 13C2-PFOA: 5.00 ng/ml 13C4-PFOS: 4.78 ng/ml 13C2-PFDA: 5.00 ng/ml	Ex. 6 Personal Privacy (PP) 100x Ex. 6 Personal Privacy (PP) Ex. 6 Personal Privacy (PP) 10x for PFHxS, PFOS and PFOA	Yes for all dilutions regardless of DF and the initial ES recovery is NOT used to quantify the final result. Results in this case are quantified using IS quant, and are therefore not ES recovery corrected per true isotope dilution technique	PFHpS [13C3-PFHxS] PFDS [13C-PFOS] PFTrDA [13C2-PFDoDA]	room temp	MDL and LOQ are reported on the form 1 (results between the MDL and LOQ are J qualified). Nominal DL for undiluted samples is approximately 0.4 ng/L for PFOS.
ALS-Kelso	**PFPeA, PFNA, 6:2FTS (ppb):** 0.01, 0.02, 0.05, 0.1, 0.25, 0.50, 1, 5, 10, 20 **PFBS, PFHpS, PFxA, PFDA, PFDoDA, PFTrDA, 8:2 FTS:** 0.02, 0.05, 0.1, 0.25, 0.50, 1, 5, 10, 20 **PFBA, PFHpA, PFOA, PFUnDA, PFTeDA, FOSA:** 0.05, 0.1, 0.25, 0.50, 1, 5, 10, ES: ES are 5.0 in all calibration standards	Phenomenex Strata-XL-AW (polymeric weak anion exchange) SPE	Per email response (2/22/2018): confirmation ions are monitored but have no evaluation criteria and are not typically provided in the lab report.	L + B standard for PFOA to set the RT window for integration . Quantitation based on linear and branched for PFHxS and PFOS. When determining the RF, the lab integrates the area under the branched and linear peaks summed together to determine the RF; an RF for the individual linear isomer and the individual branched isomer are NOT determined) The lab integrates all isomer peaks (linear and branched) for all PFAS analytes. Ex. 6 Personal Privacy (PP) **12/6 [SDG K1713183]** Linear and branched were reported for PFPeA (1x), PFNA (1x), PFBS (10x-shoulder), PFHpS (10x-shoulder) . PFHxS (100x) - if branched are present they are not resolved (broad peak), PFOS (100x), PFOA (100x-shoulder). **SDG K1712443** Ex. 6 Personal Privacy (PP) **-11/14]** Linear and branched were included in integration for PFPeA, PFOA, PFHpS, PFOS (multiple branched peaks present and all were integrated). For remaining detected results, if branched isomers are present, they are not resolved from the linear isomer. Ex. 6 Personal Privacy (PP) **11/14]** Linear and branched were included in the integration of PFPeA,PFHpS and PFOS, for remaining detected results, if the	volume	60 ml; All samples submitted in 2017 were in 60 mL containers; K1712443 included. [The sampling crew switched to 250 mL around Jan. 30, 2018]. If screening shows that results may be high, then a sub-aliquot is used. The sample containers are rinsed with methanol, and the methanol rinsate is added to the SPE cartridge to be included in the sample extraction.	All ES spiked at 5.00 ng/ml [the calibration stds and samples are prepared using the same lot of ES]	d3-MeFOSA: 5 ng/ml	Ex. 6 Personal Privacy (PP) 10x or 100x for select compounds Ex. 6 Personal Privacy (PP) **11/14:** 20x for PFOS	No, ES is not respiked when dilutions are performed. For K1713183, all samples were quantitated using IDA. If the ES was diluted out, results would be quantitated by IS method using the injection IS.	PFHpS [18O-PFHxS] PFDS [13C-PFOS] PFTrDA [13C2-PFTeDA]	4+2 C	The MRL only is reported on the form 1. (results are not reported below the MRL i.e., no J qualified data is reported). Nominal MRL for PFOS in undiluted samples is approximately 4 ng/L.
Vista	**All target compounds (pg/µl):** 0.25, 0.5, 1.0, 2.0, 5.0, 10, 50, 100 ES: ES are 12.5 pg/µl in all calibration standards	[per email response 3/7/2018] Strata X-AW 33um Polymeric Weak Anion (Phenomenex)	quant and confirmation ions used for all targets except PFBA, PFPeA, PFOSA, PFODA. EICPs are provided in reports. S/N ratios are evaluated and the pattern of branched in PFOS, PFHxS, and PFOA standards are used to determine if branched peaks should be integrated in samples. Some possible branched peaks may be excluded based on these criteria.	The technical standard for PFOS and PFHxS is used to establish retention times, but all isomers are quantified against the linear isomer only. Ex. 6 Personal Privacy (PP) **Raw [SDG 1701905]** Linear and branched were included in integration for PFHxS, PFOA, and PFOS. PFPeA: (note: a confirmation ion is not monitored for this compound) small peak in front of linear on quat ion was present but not integrated. PFBS, PFHpA, PFHpS: small peak in front of linear on quant. and conf. ion but not integrated. PFHxA: small peak in front of linear on quant ion but not present on conf. ion but not integrated. PFNA: peak before linear and larger than linear on quant and conf. ion but not integrated.	volume	250 ml [per email responses 3/7/2018: The entire sample is used and rinses the container which is then extracted. It should be noted that in some cases it is necessary to subsample. In these cases the sample container is vigrously shaken prior to the subsample. If the result is lower than expected, then the entire contents of the second bottle is used, and the container is rinsed and included in the extraction.	All ES spiked at 12.5 ng/L. [the calibration stds and samples are prepared using the same lot of ES]	All RS spiked at 12.5 ng/L. 13C6-PFDA 13C4-PFBA 13C2-4:2FTS 13C5-PFHxA 13C3-PFHxS 13C8-PFOA 13C4-PFOS 13C9-PFNA 13C7-PFUdA 13C2-FOUEA	Ex. 6 Personal Privacy (PP) 10x or 20x (PFOS is "E flagged" - not able to further dilute w/o losing ES for quant.) Ex. 6 Personal Privacy (PP) Ex. 6 Personal Privacy (PP) did not require dilution	No	PFHpS [13C2-PFOA] PFDS [13C2-PFUdA] PFTrDA [13C2-PFDoA] PFODA [13C2-PFHxDA]	<6C	DL, LOD, LOQ are reported on the form 1 (results between the DL and the LOQ are J qualified). Nominal DL for PFOS is approximately 0.4 ng/L

AECOM
250 Apollo Drive
Chelmsford, MA 01824

978-905-2100 tel
978-905-2101 fax

Memorandum

Project	North Kent Area PFAS	Page	1
Laboratory	Vista Analytical Laboratory, El Dorado Hills, CA		
Laboratory Work Number	1701905		
Analyses/Method	Per- and Polyfluoroalkyl Substances (PFAS)/Vista Lab SOP No 49, Rev 12		
Validation Level	Full		
AECOM Project Number	60560354.01 and 60556961.01		
Prepared by	Paula DiMattei		
Reviewed by	Robert Kennedy	Completed: March 6, 2018	

SUMMARY

A full validation was performed for the specified residential well water sample collected on December 6, 2017 at the North Kent site. The sample was submitted to Vista Analytical Laboratory (Vista) in El Dorado Hills, CA for analysis. Vista reported the sample under laboratory work order number 1701905.

Sample IDs
WINF1712061655JLB Ex. 6 Personal Privacy (PP) Raw]

Data validation activities were conducted with reference to:

- Vista Analytical Laboratory SOP: Preparation and Analysis for the Determination of Per- and Poly-Fluorinated Compounds (SOP No. 49, Revision 12);
- USEPA National Functional Guidelines for Organic Superfund Methods Data Review (January 2017); and
- USEPA National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016).

In the absence of method-specific information, laboratory quality control (QC) limits and/or professional judgment were used as appropriate.

REVIEW ELEMENTS

The data were evaluated based on the following review elements:

✓	Data completeness (chain-of-custody (COC)/sample integrity
✗	Holding times and sample preservation
✓	Instrument tuning
✓	Initial calibration/initial calibration and continuing calibration verification
✓	Laboratory method blanks/field blanks
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓	Laboratory control sample (LCS) results

NA	Field duplicate results
✗	Extracted internal standard results
NA	Injection internal standards
✗	Sample results/reporting issues

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. An "NA" indicates that the parameter was not included as part of this data set or was not applicable to this validation and therefore not reviewed. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

The data appear valid as reported and may be used for decision making purposes. Select data points were qualified as estimated due to nonconformances of certain QC criteria (see discussion below).

RESULTS

Data Completeness (COC)/Sample Integrity

The data package was reviewed and found to meet acceptance criteria for completeness:

- The COCs were reviewed for completeness of information relevant to the samples and requested analyses, and for signatures indicating transfer of sample custody.
- The laboratory sample login sheet(s) were reviewed for issues potentially affecting sample integrity, including the condition of sample containers upon receipt at the laboratory.
- Completeness of analyses was verified by comparing the reported results to the COC requests.

Holding Times and Sample Preservation

Sample preservation and preparation/analysis holding times were reviewed for conformance with the QC acceptance criteria. The 14-day extraction holding time was exceeded by one day for sample 1850 House St-Raw. The positive and nondetect results for all PFAS compounds in this sample were qualified as estimated (J/UJ). Qualified sample results are summarized in Table 1.

Instrument Tuning

The instrument tuning results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Initial Calibration/Initial and Continuing Calibration Verification

Calibration data were reviewed for conformance with the QC acceptance criteria to ensure that:

- the initial calibration (ICAL) percent relative standard deviation (%RSD) or correlation coefficient (r)/coefficient of determination (r^2) method acceptance criteria were met;
- the initial calibration verification standard (ICV) percent recovery (%R) acceptance criteria were met; and
- the continuing calibration verification standard (CCV) frequency and method acceptance criteria were met.

All QC acceptance criteria were met or qualification of the data was not required.

Laboratory Method Blanks/Field Blanks

Laboratory method blanks and field blanks are evaluated as to whether there are contaminants detected above the detection limit (DL). Field blanks were not submitted with this data set. Target compounds were not detected in the laboratory method blank associated with the sample in this data set.

MS/MSD Results

MS/MSD analyses were not performed on a sample in this data set. No data validation actions were taken on this basis.

LCS Results

The LCS percent recoveries were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met or qualification of the data was not required.

Field Duplicate Results

Field duplicate samples were not submitted with this data set. No data validation actions were taken on this basis.

Extracted Internal Standard Results

The extracted internal standard (IS) results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met except for the extracted IS results summarized below.

Sample ID	Extraction IS	% Recovery	QC Limits	Associated Compounds
Ex. 6 Personal Privacy (PP) -Raw	18O2-PFHxS	160	60-130	PFHxS
	13C2-PFOA	148	60-130	PFOA, PFHpS
	13C8-PFOS	153	60-130	PFOS

Samples were qualified as follows (based on NFG 2016):

Criteria	Actions[1]	
	Detected	Nondetected
%R > Upper Acceptance Limit	J	UJ
%R >10% but < Lower Acceptance Limit	J	UJ
%R <10%	See below	
<10% and S/N >10:1	J	R
<10% and S/N <10:1	R	R
[1]The PFAS method is performed using isotope dilution technique; therefore, professional judgment was applied and bias codes were not included in data qualification.		

Qualified sample results are summarized in Table 1.

Injection Internal Standard Results

The injection internal standard results were not provided since this review element is only summarized for projects requiring DoD QSM 5.1 conformance. The data are not adversely impacted.

Sample Results/Reporting Issues

If applicable, compounds detected at concentrations less than the level of quantitation (LOQ) but greater than the DL are qualified by the laboratory as estimated (J). This "J" qualifier is retained during data validation.

The result for PFOS in sample Personal Phone / Ex. 6 Raw was qualified as estimated (J) since the result exceeded the calibration range. Further dilution of this sample could not be performed since the extracted internal standard would have been diluted out at a higher dilution. EICP data did not indicate detector saturation and the result is expected to be within the linear range.

Verification of calculations was performed on a subset of the data as deemed appropriate. The calculation verification performed for the extracted internal standard was reproducible; however, the weighted polynomial calculation for a target compound in a sample was not reproducible using Excel.

QUALIFICATION ACTIONS

Sample results qualified as a result of validation actions are summarized in Table 1. All actions are described above.

ATTACHMENTS

Attachment A: Qualifier Codes and Explanations

Attachment B: Reason Codes and Explanations

Table 1 - Data Validation Summary of Qualified Data

Sample ID	Matrix	Compound	Result	LOD	LOQ	Units	Validation Qualifiers	Validation Reason
WINF1712061655JLB	WP	Perfluorooctanesulfonic acid	29800	50.4	80.7	ng/l	J	h,lc,q
WINF1712061655JLB	WP	Perfluoroheptanesulfonic acid	593	25.2	40.3	ng/l	J	h,lc
WINF1712061655JLB	WP	Perfluoroheptanoic acid	914	25.2	40.3	ng/l	J	h
WINF1712061655JLB	WP	Perfluorobutanesulfonic acid	1240	25.2	40.3	ng/l	J	h
WINF1712061655JLB	WP	Perfluorohexanesulfonic acid	4260	25.2	40.3	ng/l	J	h,lc
WINF1712061655JLB	WP	Perfluorooctanoic acid	4360	25.2	40.3	ng/l	J	h,lc
WINF1712061655JLB	WP	Perfluorooctadecanoic acid		7.56	10.1	ng/l	UJ	h
WINF1712061655JLB	WP	Perfluorooctane sulfonamide		2.52	4.03	ng/l	UJ	h
WINF1712061655JLB	WP	Perfluorotridecanoic acid		2.52	4.03	ng/l	UJ	h
WINF1712061655JLB	WP	Perfluorohexadecanoic acid		2.52	4.03	ng/l	UJ	h
WINF1712061655JLB	WP	8:2 Fluorotelomer sulfonic acid		2.52	4.03	ng/l	UJ	h
WINF1712061655JLB	WP	Perfluorotetradecanoic acid		2.52	4.03	ng/l	UJ	h
WINF1712061655JLB	WP	Perfluorononanoic acid	8.43	2.52	4.03	ng/l	J	h
WINF1712061655JLB	WP	Perfluorobutanoic acid	401	2.52	4.03	ng/l	J	h
WINF1712061655JLB	WP	Perfluorodecanesulfonic acid		2.52	4.03	ng/l	UJ	h
WINF1712061655JLB	WP	Perfluorodecanoic acid		2.52	4.03	ng/l	UJ	h
WINF1712061655JLB	WP	Perfluorododecanoic acid		2.52	4.03	ng/l	UJ	h
WINF1712061655JLB	WP	Perfluorohexanoic acid	977	2.52	4.03	ng/l	J	h
WINF1712061655JLB	WP	6:2 Fluorotelomer sulfonic acid		2.52	4.03	ng/l	UJ	h
WINF1712061655JLB	WP	Perfluoropentanoic acid	454	2.52	4.03	ng/l	J	h
WINF1712061655JLB	WP	Perfluoroundecanoic acid		2.52	4.03	ng/l	UJ	h

Attachment A

Qualifier Codes and Explanations

Qualifier	Explanation
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential low bias.
J+	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential high bias.
JN	The analyte was tentatively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Attachment B

Reason Codes and Explanations

Reason Code	Explanation
be	Equipment blank contamination
bf	Field blank contamination
bl	Laboratory blank contamination
c	Calibration issue
d	Reporting limit raised due to chromatographic interference
fd	Field duplicate RPDs
h	Holding times
i	Internal standard areas (including recovery standards)
k	Estimated Maximum Possible Concentration (EMPC)
l	LCS or OPR recoveries
lc	Extracted internal standard recovery
ld	Laboratory duplicate RPDs
lp	Laboratory control sample/laboratory control sample duplicate RPDs
m	Matrix spike recovery
md	Matrix spike/matrix spike duplicate RPDs
nb	Negative laboratory blank contamination
p	Chemical preservation issue
r	Dual column RPD
q	Quantitation issue
s	Surrogate recovery
su	Ion suppression
t	Temperature preservation issue
x	Percent solids
y	Serial dilution results
z	ICS results

AECOM
250 Apollo Drive
Chelmsford, MA 01824

978-905-2100 tel
978-905-2101 fax

Memorandum

Project	North Kent Area PFAS	Page	1
Laboratory	Eurofins-Lancaster Laboratories, Lancaster, PA		
Laboratory Work Number	1883885		
Analyses/Method	Per- and Polyfluoroalkyl Substances (PFAS)/Lab SOP TPFAS WI14355		
Validation Level	Full		
AECOM Project Number	60560354.01 and 60556961.01		
Prepared by	Paula DiMattei		
Reviewed by	Robert Kennedy	Completed: March 6, 2018	

SUMMARY

A full validation was performed for the specified residential well water sample collected on December 6, 2017 at the North Kent site. The sample was submitted to Eurofins-Lancaster Laboratories (Lancaster) in Lancaster, PA for analysis. Lancaster reported the sample under laboratory work order number 1883885.

Sample IDs
Ex. 6 Personal Privacy (PP) -IN-12/6

Data validation activities were conducted with reference to:

- Eurofins-Lancaster Laboratories' SOP TPFAS WI14355: Polyfluorinated Alkyl Substances (PFASs) in Aqueous Samples by Method 537 Revision 1.1 Modified Using LC/MS/MS (3/1/2018);
- USEPA National Functional Guidelines for Organic Superfund Methods Data Review (January 2017); and
- USEPA National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016).

In the absence of method-specific information, laboratory quality control (QC) limits and/or professional judgment were used as appropriate.

REVIEW ELEMENTS

The data were evaluated based on the following review elements:

✓	Data completeness (chain-of-custody (COC)/sample integrity
✓	Holding times and sample preservation
NA	Instrument tuning
✓	Initial calibration/initial calibration and continuing calibration verification
✓	Laboratory method blanks/field blanks
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓	Laboratory control sample (LCS)/laboratory control sample duplicate (LCSD)

	results
NA	Field duplicate results
✓	Extracted internal standard results
✓	Injection internal standards
✓	Sample results/reporting issues

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. An "NA" indicates that the parameter was not included as part of this data set or was not applicable to this validation and therefore not reviewed. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

The data appear valid as reported and may be used for decision making purposes. There were no results qualified on the basis of this data review.

RESULTS

Data Completeness (COC)/Sample Integrity

The data package was reviewed and found to meet acceptance criteria for completeness:

- The COCs were reviewed for completeness of information relevant to the samples and requested analyses, and for signatures indicating transfer of sample custody.
- The laboratory sample login sheet(s) were reviewed for issues potentially affecting sample integrity, including the condition of sample containers upon receipt at the laboratory.
- Completeness of analyses was verified by comparing the reported results to the COC requests.

Holding Times and Sample Preservation

Sample preservation and preparation/analysis holding times were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Instrument Tuning

The instrument tuning results were not reviewed as these were not provided. According to the laboratory, instrument tuning is performed after major maintenance or on an annual basis.

Initial Calibration/Initial and Continuing Calibration Verification

Calibration data were reviewed for conformance with the QC acceptance criteria to ensure that:

- the initial calibration (ICAL) percent relative standard deviation (%RSD) or correlation coefficient (r)/coefficient of determination (r^2) method acceptance criteria were met;
- the initial calibration verification standard (ICV) percent recovery (%R) acceptance criteria were met; and
- the continuing calibration verification standard (CCV) frequency and method acceptance criteria were met.

All QC acceptance criteria were met or qualification of the data was not required.

Laboratory Method Blanks/Field Blanks

Laboratory method blanks and field blanks are evaluated as to whether there are contaminants detected above the method detection limit (MDL). Field blanks were not submitted with this data set. PFDA was detected at a concentration of 0.906 ng/L in the laboratory method blank associated with sample Ex. 6 Personal Privacy (PP) IN-12/6. PFDA was not detected in this sample; therefore, qualification of the data was not required.

MS/MSD Results

MS/MSD analyses were not performed on a sample in this data set. No data validation actions were taken on this basis.

LCS/LCSD Results

The LCS and LCSD percent recoveries and relative percent differences were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Field Duplicate Results

Field duplicate samples were not submitted with this data set. No data validation actions were taken on this basis.

Extracted Internal Standard Results

The extracted internal standard results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Injection Internal Standard Results

The injection internal standard results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Sample Results/Reporting Issues

If applicable, compounds detected at concentrations less than the level of quantitation (LOQ) but greater than the MDL are qualified by the laboratory as estimated (J). This "J" qualifier is retained during data validation.

Verification of calculations was performed on a subset of the data as deemed appropriate. No discrepancies were noted.

QUALIFICATION ACTIONS

Qualification of the sample results was not required on the basis of this data review.

AECOM
250 Apollo Drive
Chelmsford, MA 01824

978-905-2100 tel
978-905-2101 fax

Memorandum

Project	North Kent Area PFAS	Page	1
Laboratory	ALS-Environmental, Kelso, WA		
Laboratory Work Number	K1713183		
Analyses/Method	Per- and Polyfluoroalkyl Substances (PFAS)/Lab SOP LCP-PFC Revision 7		
Validation Level	Full		
AECOM Project Number	60560354.01 and 60556961.01		
Prepared by	Paula DiMattei		
Reviewed by	Robert Kennedy	Completed: March 1, 2018	

SUMMARY

A full validation was performed for the indicated samples collected on December 6, 2017 at the North Kent Area site. The samples were submitted to ALS-Environmental (ALS-Kelso) in Kelso, WA for analysis. ALS-Kelso reported the samples under laboratory work order number K1713183.

Sample IDs
Ex. 6 Personal Privacy (PP) IN-12/6
FB-1850-AJC

Data validation activities were conducted with reference to:

- ALS-Kelso Laboratory SOP LCP-PFC Rev. 7: Perfluoroalkyl Substances by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS) (4/17/2017);
- USEPA National Functional Guidelines for Organic Superfund Methods Data Review (January 2017); and
- USEPA National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016).

In the absence of method-specific information, laboratory quality control (QC) limits and/or professional judgment were used as appropriate.

REVIEW ELEMENTS

The data were evaluated based on the following review elements:

✓	Data completeness (chain-of-custody (COC)/sample integrity
✓	Holding times and sample preservation
✓	Instrument tuning
✓	Initial calibration/initial calibration and continuing calibration verification
✓	Laboratory method blanks/field blanks
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results
✓	Laboratory control sample (LCS)/laboratory control sample duplicate (LCSD)

	results
NA	Field duplicate results
✓	Extracted internal standard results
✓	Injection internal standards
✓	Sample results/reporting issues

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. An "NA" indicates that the parameter was not included as part of this data set or was not applicable to this validation and therefore not reviewed. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

The data appear valid as reported and may be used for decision making purposes. There were no results qualified on the basis of this data review.

RESULTS

Data Completeness (COC)/Sample Integrity

The data package was reviewed and found to meet acceptance criteria for completeness:

- The COCs were reviewed for completeness of information relevant to the samples and requested analyses, and for signatures indicating transfer of sample custody.
- The laboratory sample login sheet(s) were reviewed for issues potentially affecting sample integrity, including the condition of sample containers upon receipt at the laboratory.
- Completeness of analyses was verified by comparing the reported results to the COC requests.

Holding Times and Sample Preservation

Sample preservation and preparation/analysis holding times were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Instrument Tuning

The instrument tuning results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Initial Calibration/Initial and Continuing Calibration Verification

Calibration data were reviewed for conformance with the QC acceptance criteria to ensure that:

- the initial calibration (ICAL) percent relative standard deviation (%RSD) or correlation coefficient (r)/coefficient of determination (r^2) method acceptance criteria were met;
- the initial calibration verification standard (ICV) percent recovery (%R) acceptance criteria were met; and
- the continuing calibration verification standard (CCV) frequency and method acceptance criteria were met.

All QC acceptance criteria were met.

Laboratory Method Blanks/Field Blanks

Laboratory method blanks and field blanks are evaluated as to whether there are contaminants detected above the reporting limit. Target compounds were not detected in the laboratory method blanks or field blanks associated with the samples in this data set.

MS/MSD Results

MS/MSD analyses were not performed on a sample in this data set. No data validation actions were taken on this basis.

LCS/LCSD Results

The LCS and LCSD percent recoveries and relative percent differences were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Field Duplicate Results

Field duplicate samples were not submitted with this data set. No data validation actions were taken on this basis.

Extracted Internal Standard Results

The extracted internal standard results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Injection Internal Standard Results

The injection internal standard results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Sample Results/Reporting Issues

Verification of calculations was performed on a subset of the data as deemed appropriate. No discrepancies were noted.

QUALIFICATION ACTIONS

Qualification of the sample results was not required on the basis of this data review.

AECOM
250 Apollo Drive
Chelmsford, MA 01824

978-905-2100 tel
978-905-2101 fax

Memorandum

Project	North Kent Area PFAS	Page	1
Laboratory	Vista Analytical Laboratory, El Dorado Hills, CA		
Laboratory Work Number	1701704		
Analyses/Method	Per- and Polyfluoroalkyl Substances (PFAS)/Vista Lab SOP No 49, Rev 12		
Validation Level	Full		
AECOM Project Number	60560354.01 and 60556961.01		
Prepared by	Paula DiMattei		
Reviewed by	Robert Kennedy	Completed: March 6, 2018	

SUMMARY

A full validation was performed for the specified residential well water samples collected on November 14, 2017 at the North Kent Area site. The samples were submitted to Vista Analytical Laboratory (Vista) in El Dorado Hills, CA for analysis. Vista reported the sample under laboratory work order number 1701704.

Sample IDs
WR1711141750JLB Ex. 6 Personal Privacy (PP) Raw]
WR1711141840 Ex. 6 Personal Privacy (PP) Raw]

Data validation activities were conducted with reference to:

- Vista Analytical Laboratory SOP: Preparation and Analysis for the Determination of Per- and Poly-Fluorinated Compounds (SOP No. 49, Revision 12);
- USEPA National Functional Guidelines for Organic Superfund Methods Data Review (January 2017); and
- USEPA National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016).

In the absence of method-specific information, laboratory quality control (QC) limits and/or professional judgment were used as appropriate.

REVIEW ELEMENTS

The data were evaluated based on the following review elements:

✓	Data completeness (chain-of-custody (COC)/sample integrity
✓	Holding times and sample preservation
✓	Instrument tuning
✓	Initial calibration/initial calibration and continuing calibration verification
✓	Laboratory method blanks/field blanks
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results

✓	Laboratory control sample (LCS) results
NA	Field duplicate results
✓	Extracted internal standard results
NA	Injection internal standards
✓	Sample results/reporting issues

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. An "NA" indicates that the parameter was not included as part of this data set or was not applicable to this validation and therefore not reviewed. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

The data appear valid as reported and may be used for decision making purposes. There were no results qualified on the basis of this data review.

RESULTS

Data Completeness (COC)/Sample Integrity

The data package was reviewed and found to meet acceptance criteria for completeness:

- The COCs were reviewed for completeness of information relevant to the samples and requested analyses, and for signatures indicating transfer of sample custody.
- The laboratory sample login sheet(s) were reviewed for issues potentially affecting sample integrity, including the condition of sample containers upon receipt at the laboratory.
- Completeness of analyses was verified by comparing the reported results to the COC requests.

Holding Times and Sample Preservation

Sample preservation and preparation/analysis holding times were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Instrument Tuning

The instrument tuning results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Initial Calibration/Initial and Continuing Calibration Verification

Calibration data were reviewed for conformance with the QC acceptance criteria to ensure that:

- the initial calibration (ICAL) percent relative standard deviation (%RSD) or correlation coefficient (r)/coefficient of determination (r^2) method acceptance criteria were met;
- the initial calibration verification standard (ICV) percent recovery (%R) acceptance criteria were met; and
- the continuing calibration verification standard (CCV) frequency and method acceptance criteria were met.

All QC acceptance criteria were met or qualification of the data was not required.

Laboratory Method Blanks/Field Blanks

Laboratory method blanks and field blanks are evaluated as to whether there are contaminants detected above the detection limit (DL). Field blanks were not submitted with this data set. Target compounds were not detected in the laboratory method blank associated with the sample in this data set.

MS/MSD Results

MS/MSD analyses were not performed on a sample in this data set. No data validation actions were taken on this basis.

LCS Results

The LCS percent recoveries were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Field Duplicate Results

Field duplicate samples were not submitted with this data set. No data validation actions were taken on this basis.

Extracted Internal Standard Results

The extracted internal standard (IS) results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Injection Internal Standard Results

The injection internal standard results were not provided since this review element is only summarized for projects requiring DoD QSM 5.1 conformance. The data are not adversely impacted.

Sample Results/Reporting Issues

If applicable, compounds detected at concentrations less than the level of quantitation (LOQ) but greater than the DL are qualified by the laboratory as estimated (J). This "J" qualifier is retained during data validation.

Verification of calculations was performed on a subset of the data as deemed appropriate. The calculation verification performed for the extracted internal standard was reproducible; however, the weighted polynomial calculation for a target compound in a sample was not reproducible using Excel.

QUALIFICATION ACTIONS

Qualification of the sample results was not required on the basis of this data review.

AECOM
250 Apollo Drive
Chelmsford, MA 01824

978-905-2100 tel
978-905-2101 fax

Memorandum

Project	North Kent Area PFAS	Page	1
Laboratory	ALS-Environmental, Kelso, WA		
Laboratory Work Number	K1712443		
Analyses/Method	Per- and Polyfluoroalkyl Substances (PFAS)/Lab SOP LCP-PFC Revision 7		
Validation Level	Full		
AECOM Project Number	60560354.01 and 60556961.01		
Prepared by	Paula DiMattei		
Reviewed by	Robert Kennedy	Completed: March 1, 2018	

SUMMARY

A full validation was performed for the specified samples collected on November 14, 2017 at the North Kent Area site. The samples were submitted to ALS-Environmental (ALS-Kelso) in Kelso, WA for analysis. ALS-Kelso reported the samples under laboratory work order number K1712443.

Sample IDs
Ex. 6 Personal Privacy (PP) N-11/14
IN-11/14
FB-11/14-C

Data validation activities were conducted with reference to:

- ALS-Kelso Laboratory SOP LCP-PFC Rev. 7: Perfluoroalkyl Substances by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS) (4/17/2017);
- USEPA National Functional Guidelines for Organic Superfund Methods Data Review (January 2017); and
- USEPA National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016).

In the absence of method-specific information, laboratory quality control (QC) limits and/or professional judgment were used as appropriate.

REVIEW ELEMENTS

The data were evaluated based on the following review elements:

✓	Data completeness (chain-of-custody (COC)/sample integrity
✓	Holding times and sample preservation
✓	Instrument tuning
✓	Initial calibration/initial calibration and continuing calibration verification
✓	Laboratory method blanks/field blanks
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results

✓	Laboratory control sample (LCS)/laboratory control sample duplicate (LCSD) results
NA	Field duplicate results
✓	Extracted internal standard results
✓	Injection internal standard results
✓	Sample results/reporting issues

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. An "NA" indicates that the parameter was not included as part of this data set or was not applicable to this validation and therefore not reviewed. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

The data appear valid as reported and may be used for decision making purposes. There were no results qualified on the basis of this data review.

RESULTS

Data Completeness (COC)/Sample Integrity

The data package was reviewed and found to meet acceptance criteria for completeness:

- The COCs were reviewed for completeness of information relevant to the samples and requested analyses, and for signatures indicating transfer of sample custody.
- The laboratory sample login sheet(s) were reviewed for issues potentially affecting sample integrity, including the condition of sample containers upon receipt at the laboratory.
- Completeness of analyses was verified by comparing the reported results to the COC requests.

Holding Times and Sample Preservation

Sample preservation and preparation/analysis holding times were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Instrument Tuning

The instrument tuning results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Initial Calibration/Initial and Continuing Calibration Verification

Calibration data were reviewed for conformance with the QC acceptance criteria to ensure that:

- the initial calibration (ICAL) percent relative standard deviation (%RSD) or correlation coefficient (r)/coefficient of determination (r^2) method acceptance criteria were met;
- the initial calibration verification standard (ICV) percent recovery (%R) acceptance criteria were met; and
- the continuing calibration verification standard (CCV) frequency and method acceptance criteria were met.

All QC acceptance criteria were met or qualification of the data was not required.

Laboratory Method Blanks/Field Blanks

Laboratory method blanks and field blanks are evaluated as to whether there are contaminants detected above the reporting limit. Target compounds were not detected in the laboratory method blanks or field blanks associated with the samples in this data set.

MS/MSD Results

MS/MSD analyses were not performed on a sample in this data set. No data validation actions were taken on this basis.

LCS/LCSD Results

The LCS and LCSD percent recoveries and relative percent differences were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Field Duplicate Results

Field duplicate samples were not submitted with this data set. No data validation actions were taken on this basis.

Extracted Internal Standard Results

The extracted internal standard results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Injection Internal Standard Results

The injection internal standard results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Sample Results/Reporting Issues

Verification of calculations was performed on a subset of the data as deemed appropriate. No discrepancies were noted.

QUALIFICATION ACTIONS

Qualification of the sample results was not required on the basis of this data review.

AECOM
250 Apollo Drive
Chelmsford, MA 01824

978-905-2100 tel
978-905-2101 fax

Memorandum

Project	North Kent Area PFAS	Page	1
Laboratory	Eurofins-Lancaster Laboratories, Lancaster, PA		
Laboratory Work Number	1876323		
Analyses/Method	Per- and Polyfluoroalkyl Substances (PFAS)/Lab SOP TPFAS WI14355		
Validation Level	Full		
AECOM Project Number	60560354.01 and 60556961.01		
Prepared by	Paula DiMattei		
Reviewed by	Robert Kennedy	Completed: March 6, 2018	

SUMMARY

A full validation was performed for the specified residential well water samples collected on November 14, 2017 at the North Kent site. The samples were submitted to Eurofins-Lancaster Laboratories (Lancaster) in Lancaster, PA for analysis. Lancaster reported the samples under laboratory work order number 1876323.

Sample IDs
Ex. 6 Personal Privacy (PP) N-11/14
IN-11/14

Data validation activities were conducted with reference to:

- Eurofins-Lancaster Laboratories' SOP TPFAS WI14355: Polyfluorinated Alkyl Substances (PFASs) in Aqueous Samples by Method 537 Revision 1.1 Modified Using LC/MS/MS (3/1/2018);
- USEPA National Functional Guidelines for Organic Superfund Methods Data Review (January 2017); and
- USEPA National Functional Guidelines for High Resolution Superfund Methods Data Review (April 2016).

In the absence of method-specific information, laboratory quality control (QC) limits and/or professional judgment were used as appropriate.

REVIEW ELEMENTS

The data were evaluated based on the following review elements:

✓	Data completeness (chain-of-custody (COC)/sample integrity
✓	Holding times and sample preservation
NA	Instrument tuning
✗	Initial calibration/initial calibration and continuing calibration verification
✓	Laboratory method blanks/field blanks
NA	Matrix spike (MS) and/or matrix spike duplicate (MSD) results

✓	Laboratory control sample (LCS)/laboratory control sample duplicate (LCSD) results
NA	Field duplicate results
✗	Extracted internal standard results
✓	Injection internal standards
✓	Sample results/reporting issues

The symbol (✓) indicates that no validation qualifiers were applied based on this parameter. An "NA" indicates that the parameter was not included as part of this data set or was not applicable to this validation and therefore not reviewed. The symbol (✗) indicates that a QC nonconformance resulted in the qualification of data. Any QC nonconformance that resulted in the qualification of data is discussed below. In addition, nonconformances or other issues that were noted during validation, but did not result in qualification of data, may be discussed for informational purposes only.

The data appear valid as reported and may be used for decision making purposes. Select data points were qualified as estimated due to nonconformances of certain QC criteria (see discussion below).

RESULTS

Data Completeness (COC)/Sample Integrity

The data package was reviewed and found to meet acceptance criteria for completeness:

- The COCs were reviewed for completeness of information relevant to the samples and requested analyses, and for signatures indicating transfer of sample custody.
- The laboratory sample login sheet(s) were reviewed for issues potentially affecting sample integrity, including the condition of sample containers upon receipt at the laboratory.
- Completeness of analyses was verified by comparing the reported results to the COC requests.

Holding Times and Sample Preservation

Sample preservation and preparation/analysis holding times were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Instrument Tuning

The instrument tuning results were not reviewed as these were not provided. According to the laboratory, instrument tuning is performed after major maintenance or on an annual basis.

Initial Calibration/Initial and Continuing Calibration Verification

Calibration data were reviewed for conformance with the QC acceptance criteria to ensure that:

- the initial calibration (ICAL) percent relative standard deviation (%RSD) or correlation coefficient (r)/coefficient of determination (r^2) method acceptance criteria were met;
- the initial calibration verification standard (ICV) percent recovery (%R) acceptance criteria were met; and
- the continuing calibration verification standard (CCV) frequency and method acceptance criteria were met.

All QC acceptance criteria were met with the following exceptions.

CCV	Compound	%R	QC Limit	Associated samples
CCV7_CAL4 12/3/2017 2:49	13C2-PFTeDA	146.9	70-130	Ex. 6 Personal Privacy (PP) -11/14
				Ex. 6 Personal Privacy (PP) -IN-11/14

Actions: (Based on NFG 2016)

Continuing calibration verification		
Criteria	Action	
	Detect	Non-detect
%D not within limits of ± 30% for target analyte or labeled compound	J	UJ

Data validation actions were applied to the results for PFTeDA in the associated samples. Qualified sample results are summarized in Table 1.

Laboratory Method Blanks/Field Blanks

Laboratory method blanks and field blanks are evaluated as to whether there are contaminants detected above the method detection limit (MDL). Field blanks were not submitted with this data set. Target compounds were not detected in the laboratory method blanks associated with the samples in this data set.

MS/MSD Results

MS/MSD analyses were not performed on a sample in this data set. No data validation actions were taken on this basis.

LCS/LCSD Results

The LCS and LCSD percent recoveries and relative percent differences were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met or qualification of the data was not required.

Field Duplicate Results

Field duplicate samples were not submitted with this data set. No data validation actions were taken on this basis.

Extracted Internal Standard Results

The extracted internal standard (IS) results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met except for the extracted IS results summarized below.

Sample ID	Extraction IS	% Recovery	QC Limits	Associated Compounds
Ex. 6 Personal Privacy (PP) -IN 11/14	13C4-PFBA	31	33-123	PFBA
	13C5-PFPeA	35	39-135	PFPeA
	13C3-PFHxS	32	34-126	PFHxS, PFHpS

Sample ID	Extraction IS	% Recovery	QC Limits	Associated Compounds
	13C4-PFHpA	33	35-126	PFHpA
	13C2-6:2 FTS	32	39-140	6:2 FTS
	13C8-PFOA	33	43-115	PFOA
	13C8-PFOSA	22	70-130	PFOSA
	13C2-8:2 FTS	38	39-137	8:2 FTS
Ex. 6 Personal Privacy (PP) -IN 11/14	13C8-PFOSA	18	70-130	PFOSA

Samples were qualified as follows (based on NFG 2016):

Criteria	Actions[1]	
	Detected	Nondetected
%R > Upper Acceptance Limit	J	UJ
%R >10% but < Lower Acceptance Limit	J	UJ
%R <10%	See below	
<10% and S/N >10:1	J	R
<10% and S/N <10:1	R	R
[1]The PFAS method is performed using isotope dilution technique; therefore, professional judgment was applied and bias codes were not included in data qualification.		

Qualified sample results are summarized in Table 1.

Injection Internal Standard Results

The injection internal standard results were reviewed for conformance with the QC acceptance criteria. All QC acceptance criteria were met.

Sample Results/Reporting Issues

If applicable, compounds detected at concentrations less than the level of quantitation (LOQ) but greater than the DL are qualified by the laboratory as estimated (J). This "J" qualifier is retained during data validation.

Verification of calculations was performed on a subset of the data as deemed appropriate. No discrepancies were noted.

QUALIFICATION ACTIONS

Sample results qualified as a result of validation actions are summarized in Table 1. All actions are described above.

ATTACHMENTS

Attachment A: Qualifier Codes and Explanations

Attachment B: Reason Codes and Explanations

Table 1 - Data Validation Summary of Qualified Data

Sample ID		Matrix	Compound	Result	MRL	Units	Validation Qualifiers	Validation Reason
Ex. 6 Personal Privacy (PP)	-IN-11/14	WP	Perfluorooctane sulfonamide		0.3	ng/l	UJ	lc
	-IN-11/14	WP	Perfluorotetradecanoic acid		0.3	ng/l	UJ	c
	N-11/14	WP	Perfluorooctane sulfonamide		0.3	ng/l	UJ	lc
	N-11/14	WP	8:2 Fluorotelomer sulfonic acid		2	ng/l	UJ	lc
	N-11/14	WP	Perfluorotetradecanoic acid		0.3	ng/l	UJ	c
	N-11/14	WP	Perfluoroheptanesulfonic acid	3	2	ng/l	J	lc
	N-11/14	WP	Perfluoroheptanoic acid	23	0.3	ng/l	J	lc
	N-11/14	WP	Perfluorobutanoic acid	11	2	ng/l	J	lc
	N-11/14	WP	Perfluorohexanesulfonic acid	160	0.4	ng/l	J	lc
	N-11/14	WP	Perfluorooctanoic acid	89	0.3	ng/l	J	lc
	N-11/14	WP	6:2 Fluorotelomer sulfonic acid		3	ng/l	UJ	lc
	N-11/14	WP	Perfluoropentanoic acid	10	0.3	ng/l	J	lc

Attachment A

Qualifier Codes and Explanations

Qualifier	Explanation
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J-	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential low bias.
J+	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample with a potential high bias.
JN	The analyte was tentatively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Attachment B

Reason Codes and Explanations

Reason Code	Explanation
be	Equipment blank contamination
bf	Field blank contamination
bl	Laboratory blank contamination
c	Calibration issue
d	Reporting limit raised due to chromatographic interference
fd	Field duplicate RPDs
h	Holding times
i	Internal standard areas (including recovery standards)
k	Estimated Maximum Possible Concentration (EMPC)
l	LCS or OPR recoveries
lc	Extracted internal standard recovery
ld	Laboratory duplicate RPDs
lp	Laboratory control sample/laboratory control sample duplicate RPDs
m	Matrix spike recovery
md	Matrix spike/matrix spike duplicate RPDs
nb	Negative laboratory blank contamination
p	Chemical preservation issue
r	Dual column RPD
q	Quantitation issue
s	Surrogate recovery
su	Ion suppression
t	Temperature preservation issue
x	Percent solids
y	Serial dilution results
z	ICS results